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(54) Title: TAPE CAST SILICON CARBIDE DUMMY WAFER (57) Abstract This invention relates to tape casting a silicon carbide slip to eventually produce a silicon carbide wafer having a thickness of between 0.5 and 1 mm and a diameter of at least 100 mm, the wafer preferably having a strength of at least 30 MPa, and a porosity wherein at least 85 % of the pores are no larger than 12 microns.		

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TAPE CAST SILICON CARBIDE DUMMY WAFER

BACKGROUND OF THE INVENTION

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The manufacture of semi-conductor devices such as diodes and transistors typically requires the deposition of dielectric materials such as polycrystalline silicon, silicon nitride and silicon dioxide on the surfaces of thin silicon wafers. The thin layer deposition of these materials involves rapid heating and cooling cycles in an electrically heated furnace (or "diffusion process tube") at temperatures typically ranging from 250 to 1000 C. When dielectric precursor gases are fed into a diffusion process tube heated to these temperatures, the gases react and deposit the dielectric reaction product on the surface of the silicon wafer.

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During the deposition step, the silicon wafers are supported in vertical or horizontal boats placed within the process tube. The wafer boat and process tube are typically made of a material which has excellent thermal shock resistance, high mechanical strength, an ability to retain its shape through a large number of heating and cooling cycles, and which does not out-gas (i.e., introduce any undesirable impurities into the atmosphere of the kiln during firing operations). One material which meets these requirements is silicon carbide. For the above-mentioned application, silicon carbide diffusion components such as boats, paddles and process tubes are typically pre-coated with the dielectric selected for deposition.

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When the silicon wafers are processed in a boat, it is naturally desirable that each wafer in the boat be exposed to identical gas concentration and temperature profiles in order to produce consistent product. However, the typical fluid dynamic situation is such that consistent profiles are found only in the middle of the boat while inconsistent profiles are often found at the ends of the boats, resulting in undesirable degrees of dielectric deposition upon the end-wafers which render them unusable.

One conventional method of mitigating this "end-effect" problem is to fill the end slots of the boat with sacrificial ("dummy") wafers made of silicon. However, it has been found that silicon wafers are expensive, extensively out-gas, warp at high process temperatures, flake particles, and have a short useful life span.

Another conventional method of mitigating the "end-effect" problem is to fill the end slots of the boat with dummy wafers made of alternative materials. For example, one investigator placed SiC-coated carbon wafers having the exact dimensions of the neighboring silicon wafers in the end slots. However, these wafers were found to break apart, contaminating the furnace with carbon particles. Another investigator proposed using CVD monolithic silicon carbide as a dummy wafer. However, this material is known to be very expensive. One prior proposal for producing silicon carbide wafers is a freeze casting approach which produces a green silicon carbide billet having a thickness of at least about 25 mm which is recrystallized and then sliced to a commercially useful thickness. However, it has been found that the freeze casting process produces significant porosity in the wafer (on the order of 40 v/o, with 15 percent of the pores larger than 25 μ m). These large pores make it difficult to completely precoat the wafer with the dielectric and make the deposition process very expensive. JP Patent Publication No. 5-283306 ("the Toshiba reference") discloses forming a commercially useful wafer by grinding down a 2 mm thick siliconized silicon carbide disc to a thickness of about 0.625 mm, and then CVD coating the disc with an alumina-silica coating. However, the grinding, siliconization and CVD steps are expensive, particularly so in the low temperature (less than 1000 C) applications where silicon infiltration is not required to prevent the oxidation of the silicon carbide.

Therefore, it is the object of the present invention to provide an inexpensive silicon carbide dummy wafer which possesses the dimensional, physical and mechanical properties

required for use in applications with temperatures less than about 1000 C.

SUMMARY OF THE INVENTION

In accordance with a preferred embodiment of the present invention, there is provided a wafer consisting essentially of silicon carbide having a thickness of between 0.5 and 1 mm, a diameter of at least 100 mm, and a strength (as measured by ring on ring biaxial flexure) of at least 30 MPa (typically between 50 and 70 MPa), the wafer having a porosity wherein at least 85% of the pores are no larger than 14 microns. Preferably, the wafer has a density of at least about 2.15 g/cc, more preferably between 2.3 and 2.4 g/cc; its porosity is between about 14% and 16%, with at least 85% of its pores being no larger than 12 microns, and at least 95% of its pores being larger than 3 microns; the average pore size is between 6 and 10 μ m, typically 8 μ m; the silicon carbide is in recrystallized form and consists of between 40 and 60 w/o grains having a size of between 2 and 5 μ m and between 40 and 60 w/o grains having a size of between about 30 and 200 microns; and, the surface of the wafer is unground.

Also in accordance with the present invention, there is provided a preferred process for making a thin, crack-free green silicon carbide sheet, comprising the steps of:

- a) forming a slip comprising a liquid carrier (preferably, water) and a ceramic powder consisting essentially of silicon carbide,
- b) tape casting the slip to produce a wet green sheet having a thickness of between 0.6 and 1.2 mm,
- c) evaporating essentially all the liquid carrier from the wet green sheet to produce a dry green sheet having a thickness which is at least 80% of the wet green sheet thickness,
- d) forming a shape from the dried green sheet to produce a silicon carbide green wafer,
- e) recrystallizing the silicon carbide green wafer to produce a recrystallized silicon carbide wafer having a

thickness of 0.5 to 1.0 mm and flatness of less than 130 um and, optionally

f) grinding the silicon carbide wafer to reduce its thickness by no more than 5%.

- 5 In preferred embodiments of this process, the silicon carbide powder consists of about 40 to 45 w/o grains having a size of between 2 and 5 um and about 38 to 42 w/o grains having a size of between about 30 and 100 microns (wherein the w/o fractions are based upon total slip weight); the water content of the
10 slip is between about 12 to 15 w/o of the slip; the total solids content (silicon carbide plus the solid portion of the binder) of the slip is between about 80 and 90 w/o of the slip; the density of the dry green sheet is at least about 2.3 g/cc; and the slip further comprises between 2 and 5 w/o of a binder
15 having a glass transition temperature of less than 22 C.

Also in accordance with the present invention, there is provided a method of using a silicon carbide dummy wafer, comprising the steps of:

- a) providing a silicon wafer diffusion boat having slots
20 for the insertion of wafers,
b) inserting into at least one slot of the silicon wafer diffusion boat a silicon carbide wafer of the present invention, said silicon carbide wafer having a coating of a dielectric material thereon,
25 c) inserting into at least one other slot a silicon wafer, and
d) depositing a dielectric material on the surface of the silicon wafer at a temperature of no more than 1000 C.

DETAILED DESCRIPTION OF THE INVENTION:

- 30 It has been found that tape casting an aqueous-based bimodal silicon carbide slip, cutting out a green wafer from the tape, and recrystallizing the green wafer provides an inexpensive, low porosity, silicon carbide wafer suitable for use as a dummy wafer.

- 35 The level of porosity produced by tape casting bimodal silicon carbide slips in accordance with the preferred process

of the present invention is typically on the order of about 14-16 v/o of the recrystallized wafer, with a pore size distribution wherein at least 95% of the pores are larger than 3 microns, at least 85% of the pores are no larger than 12
5 microns (preferably 10 microns), and the average pore size is between 4 and 8 μm , typically about 7 μm . Because the pores are both infrequent and relatively small, these wafers can be more evenly and less expensively precoated with the dielectric than can silicon carbide wafers from the freeze casting
10 process. Moreover, because the wafers do not require silicon impregnation ("siliconization"), they are less expensive than siliconized silicon carbide wafers.

In addition, because the near net shape casting described above either reduces or eliminates the need for slicing and
15 grinding, it is believed tape cast dummy wafers produced by the present invention can be produced at a significantly reduced manufacturing cost, regardless of whether the wafer is unsiliconized (for low temperature applications) or siliconized (for high temperature applications). So if an inexpensive
20 siliconized wafer is desired, the relative absence of very fine pores (i.e., pores less than 3 μm) allows for easy siliconization.

Another novel feature of the present invention is its ability to reliably provide green silicon carbide wafers of the
25 noted thickness which do not crack during drying. Typical prior art tape casting of silicon carbide was limited to using non-aqueous based slips to produce much thinner (i.e., 0.025 to 0.125 mm) green sheets. Because a dummy wafer should be the same thickness as a normal silicon wafer (i.e., about 0.625 mm
30 to 0.725 mm), the thinner wafers produced from non-aqueous based slips would not be suitable as dummy wafers. Attempts at casting larger thickness sheets from these non-aqueous based slips typically resulted in excessive cracking. Without
wishing to be tied to a theory, it is believed the excessive
35 evaporation rate of the organic solvents in these conventional slips produced a pronounced vertical disparity in the drying

rate of the relatively thick green sheet, leading to the formation of a dry skin on the top surface whose higher packing promoted cracking beneath the skin. In contrast, aqueous-based slips dry at a much slower rate. Since drying rate is slower, the evaporation profile is more uniform throughout the thickness of the sheet and formation of the undesirable dry skin is minimized. In addition, one preferred process of the present invention further enhances uniform vertical drying by heating the cast sheet from below the casting table, thereby promoting moisture removal from the bottom of the green sheet.

Moreover, it is believed that tape casting allows for reliable production of larger diameter silicon carbide dummy wafers than was previously known. In this regard, it is noted the Toshiba reference discloses a relatively small (150 mm x 150 mm x 2 mm) siliconized tile. It is also noted that one freeze casting approach which entails freeze casting a 200 mm diameter, 150 mm thick silicon carbide billet and then slicing the billet suffered from excessive cracking in the interior of a large number of the billets. In contrast, the process of the present invention allows for reliably producing suitable silicon carbide dummy wafers on the order of 200 mm and 300 mm diameters via tape casting.

Preferably, the grain size distribution of the silicon carbide grains used in the present invention is bimodal. It has been found that using a bimodal distribution produces much less shrinkage in the thin tape cast green sheet (on the order of only about 10% to 15%) than does a fine unimodal distribution (shrinkage on the order of about 85% to 90%). Since much less shrinkage is involved, the thickness of the tape is more easily controlled. Since the fine unimodal powders were the focus of the tape casting art, this advantage was not suggested in the prior art. Preferably, the bimodal SiC grain distribution comprises between about 38 and 42 w/o coarse SiC grains having a particle size ranging from 10 to 150 microns ("the coarse fraction"), and between about 40 to 45 w/o fine SiC grains

having a particle size ranging between 2 and 4 microns ("the fine fraction"). More preferably, the fine fraction comprises about 43 w/o of the SiC grain and has an average particle size of about 2-3 microns, while the coarse fraction comprises about 5 40 w/o of the SiC grain and has an average particle size of about 60 microns. In some embodiments, the fine fraction is E277, a silicon carbide powder available from Saint-Gobain/Norton Industrial Ceramics Corporation ("SG/NICC") of Worcester, MA, and the coarse fraction is F240, another silicon 10 carbide powder available from SG/NICC.

In preferred embodiments of the slip, water is present in an amount sufficient to produce a slip having from about 80 to 90 w/o solids. The slip may also include conventional additives such as deflocculents, binders and plasticizers. In 15 one preferred embodiment, the slip includes 13 w/o water, 0.01 w/o deflocculant such as sodium hydroxide, and between about 1% and about 10% (preferably between 1% and 5%) of a binder having a glass transition temperature of less than 22 C. The amount of binder used in the present invention is typically less than 20 the amount used in conventional silicon carbide tape casting, which is usually at least 10% of the slip. The use of a binder with a glass transition temperature below 22 C eliminated the need for a plasticizer in this formulation. It is believed the lower binder fraction yields a more uniform pore size 25 distribution. Typically, the silicon carbide, water and deflocculant components are mixed in a ball mill evacuated to a vacuum level of between about 27 and 30 inches Hg, the milling media is removed and the binder is added, and the entire mixture is rolled.

30 The tape casting step of the present invention is preferably accomplished by draining a reservoir containing the silicon carbide slip through a horizontally-disposed slit in the reservoir, the upper surface of slit being defined by a doctor blade and the lower surface of the slit being defined by 35 an endless belt which moves under the doctor blade. Typically, the doctor blade is positioned between about 0.4 and 1.3 mm

above the endless belt. In some embodiments, a heater is placed underneath the endless belt in order to provide a more uniform drying profile for the cast sheet. The initial thickness of the wet cast sheet thus produced is generally between about 0.4 and 1.3 mm. The cast sheet is then dried for 20-30 minutes on the heated belt, after which time the thickness of the cast sheet typically shrinks between about 10% and 15% to provide a green sheet having a density of between about 2.3 g/cc and 2.4 g/cc.

Next, the dried sheets are cut using circular punches to form green silicon carbide wafers having a diameter of between 100 and 300 mm and a thickness of between about 0.5 and 1.1 mm, preferably between 0.5 and 1 mm, more preferably between about 0.625 and 0.725 mm. When the wafers are in the range of about 0.625 mm and 0.725 mm, they do not need to be surface ground.

In some embodiments, the scrap material from the dried cut sheet is recycled. Preferably, the scrap is completely dispersed by milling overnight in 25 w/o deionized water; the resulting slip is then mixed with virgin silicon carbide powders and additives; remilled; and water is added to the slip to bring its viscosity to about 30,000 cps at 0.6 rpm. This recycled slip can then be used to make more green sheets, thereby essentially eliminating waste of the silicon carbide raw material.

In some embodiments, the cast wafers are heat treated prior to recrystallization in order to partially remove the binder. It has been found that partial removal of the binder (i.e., removal of about 15% to 25%) yields a fine pore structure in the sintered body which is advantageous for pre-coating. It is believed the partial burnout produces small diameter vapor "escape routes" for the remaining binder, thus preventing the creation of larger sized pores during the recrystallization process. In preferred embodiments, this entails exposing the cast wafers to air at a temperature of 220°C and a pressure of 5 inches Hg for 8 hours.

Next, the dried green wafers are recrystallized. Recrystallization establishes strength-enhancing neck growth

between the SiC grains without substantial densification. It is generally carried out at about 1900-1950°C under a vacuum of about 0.6 torr in an Ar atmosphere. In preferred embodiments, the wafers are recrystallized at 1950°C and 0.6 torr in argon.

5 In some embodiments, the green wafers are stacked between surface ground silicon carbide plates in order to provide flatness.

A recrystallized wafer produced in accordance with the bimodal embodiment of the present invention typically exhibits
10 a bulk density (as measured by mercury intrusion porosimetry) of at least about 2.15 g/cc, and preferably between 2.3 and 2.4 g/cc, a total porosity of about 14-16%, an average pore size of between 4 and 8 μm , with a pore size distribution wherein at least 95% of the pores are larger than 3 microns and at least
15 85% of the pores are no larger than 12 microns (preferably 10 microns). In preferred embodiments using a bimodal grain distribution, the fine fraction grains average about 2 to 5 microns and the coarse fraction grains average about 30 to 100 microns. It has a measure of flatness of no more than about
20 130 μm across a diameter of about 200 mm. Its strength (as measured by ring on ring biaxial flexure) is typically between about 50 MPa and about 70 MPa.

Recrystallized silicon carbide dummy wafers having diameters of at least 100 μm , preferably between about 150 and
25 about 200 mm; thicknesses of between about 0.5 and about 1 mm, and preferably about between about 0.625 mm and 0.725 mm; and flatnesses of between about 50 and about 130 microns, preferably less than about 100 microns, are obtainable in accordance with this embodiment.

30 If desired, an additional firing step may be undertaken to make the wafer resistant to gas or liquid attack in high temperature applications. This typically involves either impregnating the recrystallized wafer with silicon to eliminate porosity and/or CVD coating it with an impermeable ceramic such
35 as silicon carbide. If siliconizing is selected, it may be carried out in accordance with US Patent No. 3,951,587 ("the

Alliegro patent"), the specification of which is incorporated herein by reference. Therefore, in accordance with the present invention, there is provided a wafer consisting essentially of silicon carbide having a thickness of between 0.5 and 1 mm, a diameter of at least 100 mm, the wafer being infiltrated with silicon so that the silicon is present as silicon pockets and comprises about 14 to 16 v/o of the wafer, and wherein at least 85% of the silicon pockets are no larger than 10 microns (preferably 8 microns) and at least 95% of the silicon pockets are larger than 3 microns. There is also provided a method of using this siliconized silicon carbide wafer, comprising the steps of:

- a) providing a silicon wafer diffusion boat having slots for the insertion of wafers,
- b) inserting into the slot of the silicon wafer diffusion boat a silicon carbide dummy wafer as described in this paragraph, said dummy wafer having a coating of CVD silicon carbide thereon,
- c) inserting a silicon wafer into another slot in the boat, and
- d) oxidizing the surface of the silicon wafer at a temperature of at least 1000 C.

If CVD coating with silicon carbide is selected, it may be carried out by any conventional CVD SiC method. Likewise, the silicon carbide wafer of the present invention may be coated with a dielectric material such as polycrystalline silicon, silicon nitride, or silicon dioxide.

Conventional sandblasting of the siliconized SiC wafer can remove excess free silicon that has exuded to the surface due to the volume expansion of silicon on solidification. Because these wafers possess high strength, they do not break when subjected to sandblasting.

The novel recrystallized silicon carbide wafers of the present invention are preferably used as dummy wafers in silicon wafer manufacturing. However, they may also find application as rigid discs in computer hard drives; as

substrates for other micro-electronic applications including acting as setters in single wafer processing and plasma etching; as substrates for flat panel LCD displays; or as baffle plates in wafer boats.

5 Also in accordance with the present invention, there is provided a preferred method of single wafer processing, comprising the steps of:

- 10 a) providing a silicon carbide disk of the present invention (preferably having a diameter of at least 200 mm and more preferably at least 300 mm) in a substantially horizontal position, and
- b) placing a silicon wafer (preferably having a diameter of at least 200 mm and more preferably at least 300 mm) upon the silicon carbide disc, and
- 15 c) heating the silicon wafer at a rate of at least 100 C per second.

Also in accordance with the present invention, there is provided a method of cleaning single wafer processing chambers, comprising the steps of:

- 20 a) providing a susceptor in a processing chamber,
- b) placing a silicon wafer upon the susceptor,
- c) processing the silicon wafer,
- d) removing the silicon wafer,
- e) placing a silicon carbide disk of the present invention
- 25 (preferably having a diameter of at least 200 mm and more preferably at least 300 mm) over the susceptor, and
- f) in-situ cleaning the processing chamber by exposure to free radicals.

Also in accordance with the present invention, there is provided a method of flat panel display processing, comprising the steps of:

- 30 a) providing a silicon carbide plate of the present invention (preferably having a length of at least 165 mm and a width of at least 265 mm) in a substantially
- 35 horizontal position, and

b) placing a flat glass plate (preferably having a length and a width of at least 100 mm) upon the silicon carbide plate, and

5 c) processing the flat glass plate by oxidation, dielectric deposition and/or diffusion at a temperature of no more than 800 C.

Also in accordance with the present invention, there is provided a method of plasma etching silicon wafers, comprising the steps of:

- 10 a) providing a silicon wafer having a predetermined diameter of at least 200 mm,
b) placing a silicon carbide ring of the present invention (having an inner diameter essentially equal to the predetermined diameter of the silicon wafer) around the
15 silicon wafer, and
c) plasma etching (preferably, dry metal plasma etching) the silicon wafer.

Other contemplated uses of the silicon carbide wafers of the present invention (which could exploit an expected low
20 pressure drop across the wafer) include gas burner plates, composite substrates and filters.

For the purposes of the present invention, "v/o" refers to a volume percent, "w/o" refers to a weight percent. In addition, the term "flatness" is considered to be the total
25 spread between the minimum and maximum deflection from a flat granite plate.

EXAMPLE I

A bimodal powder consisting of about 42 w/o fine silicon carbide and about 39 w/o coarse silicon carbide and a
30 deflocculant were mixed with about 8 w/o deionized water, 4 w/o latex binder and 6 w/o plasticizer (PPG). The resulting slip was milled overnight under vacuum. The viscosity of the slip was found to be about 30,000 to 35,000 cps at 0.6 rpm.

Bubbles were observed in the milled slip. These bubbles
35 are believed to result in small pin holes in the green tape casting.

A conventional tape casting table was used to cast the slip. The unit included a drive control, mylar carrier, slurry reservoir, doctor blade, supporting table, drying unit and take-up drum. The unit also had an electric heater below the
5 table so that moisture in the slip was removed from the bottom of the tape upwards, thereby preventing a skin from forming at the surface of the tape.

Eight foot lengths of slip were tape cast at 300 mm per minute in widths varying between 150 and 300 mm, and at a blade
10 height of 1.25 mm. The cast tape was then dried for about 1 hour at 30°C before removal from the table. The tapes were subsequently allowed to dry overnight at room temperature to enhance their green strength. The drying resulted in the tapes shrinking essentially exclusively in the thickness dimension so
15 that the dried thickness was about 0.94 mm. Wafers were cut from the dried tape in diameters of about 100 mm, 150 mm and 200 mm.

In preparation for recrystallization, the green wafers were stacked between horizontal dense silicon carbide plates to
20 form a column, with graphite paper inserted between each side of the green wafer to prevent sticking. The wafers were then recrystallized at 1950°C and 900 mtorr.

The pin holes observed in the cast tape were not observed in the recrystallized wafers. However, some of the wafers were
25 found to be bent around the edges and most displayed an imprint on their surfaces. The cause of the bending was believed to be the silicon carbide plates sliding during recrystallization, with the graphite paper providing lubrication for the sliding.

The cause of the imprint was believed to be the thermal
30 decomposition of the graphite paper.

Oxidation of selected recrystallized wafers having the imprint did not appreciably remove the imprint. The imprint, however, was not deleterious to the end product.

EXAMPLE II

35 In this example, scrap tape from experiments conducted in substantial accordance with Example I was used as a starting

material. The scrap tape was completely dispersed by milling overnight in 25 w/o deionized water. The resulting slip was then mixed with the silicon carbide powders, deflocculant, binder, and plasticizer as above, and then milled overnight under vacuum in a nylon jug. Water was then added to the slip to bring its viscosity to about 30,000 cps at 0.6 rpm. Slip comprising up to 50 w/o scrap produced high quality tapes.

EXAMPLE III

This example attempted to reduce the pin holes observed in the green bodies of Example I. Since close observation of the slip revealed small, oily bubbles, it was believed the pin holes were the result of incomplete dissolution of either the latex binder or plasticizer. Since the plasticizer (polypropylene glycol) is prone to coagulation at the high shear rates found in milling, it was proposed that the plasticizer be blended with the slip after the water has coated the particles.

Accordingly, a new slip was prepared by vacuum milling the recycled slip of Example II, silicon carbide power, and deflocculant; removing the milling balls; adding the pre-mixed binder and plasticizer, and then vacuum rolling the mixture in a nylon jug overnight. The resulting slip had a significantly lower viscosity (about 9900 cps at 0.6 rpm). Upon casting, it was observed that the frequency of pin holes was sharply reduced but not eliminated.

EXAMPLE IV

This example was performed in substantial accordance with Example I, except that a separate burnout step was incorporated before the recrystallizing step.

The green wafers produced in substantial accordance with Example I were set on recrystallized silicon carbide plates and the plates were stacked with silicon carbide spacers in between to form columns. These columns were exposed to air at a temperature of 200°C and a vacuum of 5 inches Hg for 6 hours to promote burnout of the plasticizer. This treatment decomposed the plasticizer and cured the binder. The removal of the

plasticizer eliminated wafer sticking and the subsequent need for graphite paper and hence the sliding problem described in Example I.

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EXAMPLE V

Green wafers cast and subject to plasticizer burnout as in Example IV were stacked in groups of ten between a top and a bottom surface ground SiC plate, but without any graphite paper inserted therebetween. The wafers were then recrystallized as in Example I.

It appears that the wafers did not slide during recrystallization. Their measure of flatness was less than about 0.005". In addition, sticking between the wafers was minimal, as the wafers could be easily pried apart. Breakage from prying apart stuck wafers was only about 0-6%.

EXAMPLE VI

A bimodal powder consisting of about 43 w/o fine silicon carbide and about 40 w/o coarse silicon carbide and a deflocculant were mixed with about 13 w/o deionized water and 0.01 w/o NaOH solution. The slip was ball milled under vacuum (25 in. Hg) for 12 hours at 200 rpm. About 3 w/o of a latex emulsion binder was added and the slip was mixed without vacuum for 2 hours at 25 rpm. A trace amount of a surfactant (0.1 w/o) was also added to improve the wetting behavior of the slip on the mylar carrier. The total solids content of the slip was between 85 w/o and 90 w/o and the viscosity was about 20,000 cps at 0.6 rpm.

The tape casting table described in Example I was used to tape cast the slip. Tapes with a green thickness of 0.5-1.0 mm were obtained with the following casting parameters: a reservoir height of 10 to 30 mm, carrier speed of 0.4 to 1.1 m/min; doctor blade height of 0.4 - 0.7 mm; and under bed heating temperature of 35-45 C. Depending upon the thickness, the tapes required a total drying time of 20 - 40 minutes, resulting in a 10-15% shrinkage during drying. The green tape

was collected from the exit end of the table and sectioned into 1 meter long sheets. Wafers were cut from the green tape in diameters of 100-300 mm. The green wafers were subject to binder burnout at 200 C for 8 hours in air, and recrystallized
5 as in Example V. The densities of the recrystallized wafers were between 2.3 and 2.4 g/cc.

The recrystallized wafers were then siliconized and sandblasted through conventional means. The densities of the resulting wafers were found to be about between 2.91 and 2.98
10 g/cc.

Preliminary studies of pressing the flexible green tapes with a hydraulic press at 8000 psi followed by binder burnout as in Example IV and recrystallization as in Example V have demonstrated an increase in density up to 2.55 g/cc while the
15 average pore size was reduced to 6 microns. Similar results were obtained by other compaction methods such as cipping at 30,000 psi or passing the tape through a rolling operation.

Although the above SiC ceramic has utility as a dummy
20 wafer, there is also interest in making substrates for the deposition of diamond films to be used in thermally enhanced plastic packages for semiconductor devices. For such applications, the SiC substrate must be strong and thin. It is believed that, in order to meet these requirements, the SiC
25 ceramic should have a thickness of between about 0.1 and 0.6 um, and its strength should be at least 80 MPa. Unfortunately, the 0.5 mm thick tape cast SiC wafers described above in Examples I-VI, which were found to have a D_{15} pore diameter of about 3 um, a D_{85} pore diameter of 12 um, and a biaxial
30 flexural strength of only about 50-70 MPa, do not meet these requirements. Therefore, there is a need for a tape cast recrystallized silicon carbide ceramic (preferably having a thickness of between 0.1 and 0.6 mm) having a strength of at least 80 MPa.

EXAMPLE VII

In this example, the effect of solely using a fine silicon carbide powder was examined. In particular, the procedure of Example I above were substantially followed, except that the coarse silicon carbide fraction was eliminated.

5 The resulting tape exhibited significant shrinkage and cracking during drying.

EXAMPLE VIII

In this example, both the fine/coarse ratio of the SiC powder and the sintering temperature were modified from those
10 of the above Examples (which had about 52 wt% fines and 48 wt% coarse particles, and were sintered at about 1950°C).

The procedure of Example VI above was substantially followed, except that the three different fine/coarse SiC powder ratios (35/65, 50/50, and 65/35) and three different
15 sintering temperatures (1700°C, 1850°C, and 2000°C) were used.

In particular, the coarse grain fraction was first sieved through a 50 um screen to remove its coarse tail. Initial water contents of 15% was used for the 35% fines formulation, while 12% was used for the remaining two formulations. Deflocculation
20 curves were prepared for each formulation in according with ISO manufacturing procedure, and the optimal deflocculation points were determined. Next, 1000 gram batches of the three formulations were prepared. The amount of B-1035 binder used was based upon the surface area of the grains, and a
25 conventional non-ionic surfactant was used at a level of 2 wt% of the binder. The optimal water fraction for each formulation was determined by making incremental additions until a slip viscosity of 25 (dial) at 0.6 rpm (as measured by a Brookfield viscometer with a LV-2 spindle) was attained. The
30 solids loadings for these formulations were all between 85 wt% and about 87 wt% (66-68 vol%).

The three formulations were de-aired in a vacuum bell jar at 15"Hg for 15 minutes. The batches were tape cast at a carrier speed of 20"/minute at a blade height of 0.016" and a
35 temperature of 150°F. The slurry was poured manually into the reservoir, maintaining a reservoir height of 1.0 cm. The

average dried green tape thickness for each batch was between about 0.128 and 0.132 inches, and was determined by cutting out 100 mm diameter green wafers and measuring the thickness of the four quadrants.

5 The wafers from each batch were cross-rolled (i.e., rolled, rotated 90°, and rolled again) through spaced 15 cm diameter, hardened steel, primary rollers having a roller gap of 0.005". The significant elastic behavior of the wafers typically results in only about 5-10% compaction from the green
10 thickness. The average rolled wafer thickness for each batch was determined by measuring the thickness of the same wafers at the four quadrants.

 The wafers were loaded into the sintering furnace on SiC setter plates. Binder burnout was conducted at a ramp rate
15 0.2°C/minute and an 8 hour soak at 240°C. The wafers from each batch were divided into three groups and then sintered at the desired temperature. Each of the sintering cycles started with a 3°C per minute ramp to 500°C, with an intermediate soak for 1 hour to complete the burnout cycle. The runs were then ramped
20 at a rate of 5°C/minute to the final soak temperature with a soak time of three hours. Each of the three runs was conducted under Argon at a pressure of 0.6 torr for the entire cycle.

 Sintered density and pore size distribution was measured by mercury intrusion porosimetry. The biaxial flexural
25 strengths were measured by a ring-on-ring configuration.

 The sintered densities of the resulting materials are shown in Table I.

Table I
Sintered Densities (g/cc)

	<u>35%fine/65% coarse</u>	<u>50%fine/50%coarse</u>	<u>65%fine/35%coarse</u>
1700°C	2.34	2.31	2.21
1850°C	2.54	2.41	2.32
2000°C	2.51	2.49	2.36

5 Examination of Table I reveals that densities increase both with increasing coarse fraction and increasing sintering temperature. Increasing the fines content of the formulation requires the use of additional binder and liquid during green body formation in order to prevent cracking. The resulting
10 decreased density of the green body leads to a decreased density in the fired body. Similarly, it is believed that increasing the sintering temperature produces more sintering activity, grain coarsening, and neck formation, and therefore higher densities.

15 The D₅₀ pore diameters of the resulting materials are shown in Table II.

Table II

	D ₅₀ pore Diameters (μm)		
	<u>35%fine/65% coarse</u>	<u>50%fine/50%coarse</u>	<u>65%fine/35%coarse</u>
1700°C	2.2	1.7	1.8
1850°C	6.0	4.0	3.3
2000°C	11.0	10.7	7.9

20 Examination of this table reveals that pore diameter increases with sintering temperature and coarse fraction.

The characteristic flexural strengths of the resulting materials, as determined by Weibull analysis, are shown in Table III.

Table III
Flexural Strength (MPa)

	<u>35%fine/65% coarse</u>	<u>50%fine/50%coarse</u>	<u>65%fine/35%coarse</u>
1700°C	47.7	95.0	103.5
1850°C	31.0	65.4	81.3
2000°C	47.8	49.5	47.1

5 Examination of the above table reveals the surprising result that lower sintering temperatures and higher fine fractions lead to higher strength wafers.

The results are surprising in two respects. First, it is clear that different phenomenon are occurring at different fine/coarse fractions and different temperatures. In particular, in the Table III studies in which the SiC powder was kept constant at 35% fines/ 65% coarse, increasing the sintering temperature from 1850°C to 2000°C increases the flexural strength from 31 MPa to 47.8 MPa. It is believed, in relatively coarse formulations, the extent of inter-grain necking (which increased with sintering temperature) is the dominant factor. In contrast, in the studies in which the powder was kept constant at 65% fines/ 35% coarse, increasing the sintering temperature leads to lower strength. It is believed that, in relatively fine powder formulations, pore size (which increases with sintering temperature) becomes the critical flaw.

Similarly, in the studies in which the sintering temperature was kept constant at 2000°C, increasing the fine fraction from 35% to 65% had relatively no effect on the flexural strength. It is believed that, in high fired embodiments, the large pores created by the grain coarsening are the strength limiting feature. In contrast, in the studies in which the sintering temperature was kept constant at 1700°C increasing the fine fraction from 35% to 65% increased the flexural strength from 47.7 to 103.5 MPa.

In sum, these studies do not follow the traditional expectations that simply increasing sintering temperature and decreasing grain size leads to increased strength.

Second, the strengths recorded in Table III do not correlate well with traditional factors typically correlating positively with strength (i.e., density and D_{50} pore diameter). For example, the example having 35% fines and sintered at 1700°C possessed a fairly low strength (47.7 MPa) despite its relatively small 2.2 μm D_{50} pore diameter. Also, the fired bodies having the highest densities (i.e., above 2.5 g/cc) recorded some of the lowest strengths (31.1 MPa and 47.8 MPa).

The sintered bodies were also analyzed for D_{85} pore diameter, which represents the size which exceeds exactly 85% of the pores. The D_{85} pore diameter of the examples are shown in Table IV below, together with the corresponding flexural strength in parentheses:

Table IV

	D_{85} Pore Diameters, in μm (flexural strengths in parentheses)		
	<u>35%fine/65% coarse</u>	<u>50%fine/50%coarse</u>	<u>65%fine/35%coarse</u>
1700°C	5.7 (47.7MPa)	2.0 (95.0 MPa)	2.1 (103.5 MPa)
1850°C	9.0 (31.0 MPa)	4.8 (65.4 MPa)	3.9 (81.3 MPa)
2000°C	12.5 (47.8 MPa)	12.2 (49.5 MPa)	9.8 (47.1 MPa)

Comparison of the D_{85} pore diameters with the flexural strengths reveals a very strong correlation between D_{85} pore diameter and flexural strength. For example, only examples having a D_{85} pore diameter of no more than 4 μm possess a flexural strength greater than 80 MPa, and the examples with the smallest D_{85} pore diameters (about 2 μm) have the largest flexural strength. Further, the low strength (47.7 MPa) of the example having the 2.2 μm D_{50} pore diameter is now explained by its unexpectedly larger D_{85} pore size (5.7 μm).

Therefore, it is now clear that providing a D_{85} pore diameter of less than 4 μm , and more preferably less than 2.5

um is critical to providing a flexural strength of at least 80 MPa, and preferably at least 95 MPa, and that using either :

- i) at least 50% fines (preferably at least 60%) and sintering at no more than 1750°C , or
- 5 ii) at least 60% fines and sintering at no more than 1850°C (preferably 1750°C)

is also critical to producing these low D_{85} pore diameter values which produce the high strength SiC wafers.

Therefore, in accordance with the present invention, there
10 is provided a ceramic wafer consisting essentially of recrystallized silicon carbide grain and having a D_{85} pore diameter of no more than 4 um, and a biaxial flexure strength of at least 80 MPa, wherein between about 50 wt% and 70 wt% of the silicon carbide comprises fine grains having a grain size
15 of no more than 5 um, and between 30 wt% and 50 wt% of the SiC comprises coarse grains having a grain size of at least 20 um.

In some embodiments, the wafer has a density of no more than 2.33 g/cc, preferably a D_{85} pore diameter of no more than
20 3 um, and a strength of at least 90 MPa.

In some embodiments, the wafer has a density of no more than 2.25 g/cc and a D_{85} pore diameter of no more than 2.5 um, a strength of at least 100 MPa, and between about 60 wt% and 70 wt% of the silicon carbide comprises fine grains having a
25 grain size of no more than 5 um.

In some embodiments, the ceramic has a thickness of between 0.1 mm and 0.3 mm, is preferably unground, and more preferably has a D_{50} pore diameter of at least about 1 um.

In some embodiments, between 30 wt% and 50 wt% of the SiC
30 comprises coarse grains having a grain size of at least 30 um.

Also in accordance with the present invention, there is provided a process for making high strength, tape cast SiC wafers, comprising the steps of:

- a) providing a formulation consisting essentially of
35 silicon carbide powder and water, wherein:

- i) between 50 wt% and 70 wt% of the SiC powder has a particle size of no more than 5 μm , and
- ii) between 30 wt% and 50 wt% of the SiC powder has a particle size of at least 30 μm ,
- 5 b) tape casting the formulation to produce a tape cast body,
- c) drying the tape cast body to produce a green body having a thickness of between about 0.1 mm and 0.6 mm, and
- 10 d) sintering the green body at a temperature of no more than 1850°C,
- to form a recrystallized silicon carbide wafer having a density of no more than 2.35 g/cc and a strength of at least 80 MPa.
- 15 In some embodiments, the sintering temperature is no more than 1750°C, and the recrystallized silicon carbide wafer has a D_{85} pore size of no more than 2.5 μm and a strength of at least 90 MPa.
- In some embodiments, the SiC powder comprises at least 60
- 20 wt% SiC particles having a size of no more than 5 μm , and the recrystallized silicon carbide wafer has a D_{85} pore diameter of no more than 3 μm .
- In some embodiments, the sintering temperature is no more than 1750°C, the SiC powder comprises at least 60 wt% SiC
- 25 particles having a size of no more than 5 μm , and the recrystallized silicon carbide wafer has D_{85} pore size of no more than 2.5 μm , a density of no more than 2.25 g/cc and a strength of at least 100 MPa.
- In some embodiments, the sintering temperature is no more
- 30 than 1700°C, the SiC powder comprises at least 65 wt% SiC particles having a size of no more than 5 μm , and the recrystallized silicon carbide wafer has a strength of at least 103 MPa.
- In some embodiments, the process includes the additional
- 35 step of compacting the tape cast body to reduce its thickness by at least 5% (preferably at least 10%) prior to sintering.

EXAMPLE IX

This example examines the effect of roll compacting the tape cast green SiC wafer.

Example VIII above was substantially followed, except that
5 the formulation comprised 57% fine/43% coarse SiC particles, the sintering temperature was 1850°C, and the dried tape cast wafer was not subjected to roll compaction.

The resulting wafers had a density of about 2.36 g/cc, a
D₈₅ pore diameter of about 8.5 um, and a flexural strength of
10 only about 35-45 MPa.

This example can be usefully compared with the 50% fine and 65% fine embodiments of Example VIII above which were roll compacted and sintered at 1850°C, and possessed flexural strengths of 65.4 MPa and 81.5 MPa respectively. Since each of
15 the previous examples (one of which had slightly less fines and one of which had slightly more fines) had twice the flexural strength, it is clear that roll compacting the tape cast wafer had a significantly positive effect upon the flexural strength of the recrystallized wafer. Without wishing to be tied to a
20 theory, it is believed that roll compacting has the effect of decreasing the interparticle spacing to promote more effective neck formation during recrystallization.

Therefore, also in accordance with the present invention, there is provided a process comprising the steps of:

- 25 a) providing a formulation consisting essentially of a ceramic powder, binder and water,
b) tape casting the formulation to produce a tape cast body,
c) drying the tape cast body to produce a green body,
30 d) compacting the green body to reduce its thickness by at least 10%, and
d) sintering the green body.

For the purposes of the present invention, pore diameter is determined by mercury intrusion porosimetry.

35

I Claim:

1. A wafer consisting essentially of silicon carbide having a thickness of between 0.5 and 1 mm, a diameter of at least 100 mm, and a strength of at least 30 MPa, the wafer having a porosity wherein at least 85% of the pores are no larger than 12 microns.
2. The wafer of claim 1 having a density of at least about 2.15 g/cc.
3. The wafer of claim 1 wherein at least 85% of the pores are no larger than 10 microns.
4. The wafer of claim 1 wherein at least 95% of the pores are larger than 3 microns.
5. The wafer of claim 1 wherein the silicon carbide is in recrystallized form and consists of grains having a size of between 2 and 5 μm in the range of 40 to 60 w/o of the wafer and grains having a size of between about 30 and 200 microns in the range of between 40 and 60 w/o of the wafer.
6. The wafer of claim 1 wherein the silicon carbide is in recrystallized form and consists of grains having a size of between 2 and 5 μm in the range of between 50 and 55 w/o of the wafer and between grains having a size of between about 30 and 100 microns in the range of between 45 and 50 w/o of the wafer.
7. The wafer of claim 1 wherein the surface of the wafer is unground.
8. The wafer of claim 1 coated with a dielectric material selected from the group consisting of polycrystalline silicon, silicon nitride and silicon dioxide.
9. A process for making a thin, crack-free green silicon carbide sheet, comprising the steps of:
 - a) forming a slip comprising a carrier liquid and a ceramic powder consisting essentially of silicon carbide,
 - b) tape casting the slip to produce a wet green sheet having a thickness of between 0.4 and 1.3 mm.
10. The process of claim 9 further comprising the step of:

c) evaporating essentially all the carrier liquid from the wet green sheet to produce a dry green sheet having a thickness which is at least 80% of the wet green sheet.

11. The process of claim 9 wherein the silicon carbide powder consists of about 43 w/o grains having a size of between 2 and 5 um and about 40 w/o grains having a size of between about 30 and 100 microns.

12. The process of claim 9 wherein the carrier liquid is water and total water content of the slip is between about 10 and 15 w/o of the slip.

13. The process of claim 12 wherein the silicon carbide solids content of the slip is between about 80 and 90 w/o of the slip.

14. The process of claim 13 wherein the density of the green sheet is at least 2.3 g/cc.

15. The process of claim 9 wherein the slip further comprises between 3 and 5 w/o of a binder having a glass transition temperature of less than 22 C.

16. The process of claim 10 further comprising the steps of:

d) forming a shape from the dried green sheet to produce a silicon carbide green wafer, and

e) recrystallizing the silicon carbide green wafer to produce an unground recrystallized silicon carbide wafer having a thickness of between 0.625 mm and 0.725 mm and a measure of flatness of less than 130 microns.

17. The process of claim 16 further comprising the step of:

f) grinding the silicon carbide wafer to reduce its thickness by no more than 5%.

18. A process for making a silicon carbide body, comprising the steps of:

a) forming a slip comprising water and a ceramic powder consisting essentially of silicon carbide, and

b) tape casting the slip to produce a wet green sheet,

c) drying the wet green sheet to produce an dry green sheet having a thickness of between about 0.625 and 0.725 mm,

- d) forming a shape having a diameter of at least 200 mm from the dry green sheet to form a green body, and
e) recrystallizing the green body to form a silicon carbide body having a diameter of at least 200 mm and a flatness of less than 130 microns.
- 5
20. An unground wafer consisting essentially of silicon carbide, the wafer having a thickness of between 0.5 and 1 mm and a diameter of at least 100 mm.
21. The unground wafer of claim 20 having a density of at least about 2.15 g/cc.
- 10
22. The wafer of claim 20 having a strength of at least 30 MPa.
23. The wafer of claim 20 wherein the silicon carbide is in recrystallized form and consists of grains having a size of between 2 and 5 um in the range of about 50 to 55 w/o of the wafer and grains having a size of between about 30 and 100 microns in the range of about 45 to 50 w/o of the wafer.
- 15
24. The wafer of claim 20 having a porosity wherein at least 85% of the pores are no larger than 10 microns, the wafer being coated with a dielectric material selected from the group consisting of polycrystalline silicon, silicon nitride and silicon dioxide.
- 20
25. A method of using a silicon carbide dummy wafer, comprising the steps of:
- a) providing a silicon wafer diffusion boat having slots for the insertion of wafers,
- 25
- b) inserting into a slot of the silicon wafer diffusion boat a silicon carbide wafer as described in claim 1, said dummy wafer having a coating of a dielectric material thereon
- 30
- c) inserting a silicon wafer into another slot in the boat, and
- d) depositing a dielectric material on the surface of the silicon wafer at a temperature of no more than 1000 C.
26. A wafer consisting essentially of silicon carbide having a thickness of between 0.5 and 1 mm, a diameter of at least 100 mm, the wafer being infiltrated with silicon so that the
- 35

silicon is present as silicon pockets, and wherein at least 85% of the silicon pockets are no larger than 10 microns and at least 95% of the silicon pockets are larger than 3 microns.

27. A method of using a silicon carbide dummy wafer, comprising
5 the steps of:

- a) providing a silicon wafer diffusion boat having slots for the insertion of wafers,
- b) inserting into the slot of the silicon wafer diffusion boat a silicon carbide dummy wafer as described in claim
10 26, said dummy wafer having a coating of CVD silicon carbide thereon,
- c) inserting a silicon wafer into another slot in the boat, and
- d) oxidizing the surface of the silicon wafer at a
15 temperature of at least 1000 C.

28. A ceramic wafer having a thickness of between 0.1 and 0.6 mm and consisting essentially of recrystallized silicon carbide grain, the wafer having a D_{85} pore diameter of no more than 4 μm , and a biaxial flexure strength of at least 80 MPa,
20 wherein between about 50 wt% and 70 wt% of the silicon carbide comprises fine grains having a grain size of no more than 5 μm , and between 30 wt% and 50 wt% of the silicon carbide comprises coarse grains having a grain size of at least 20 μm .

25 29. The ceramic of claim 28 having a density of no more than 2.33 g/cc.

30. The ceramic of claim 29 having a D_{85} pore diameter of no more than 3 μm , and a strength of at least 90 MPa.

31. The ceramic of claim 30 having a density of no more than
30 2.25 g/cc and a D_{85} pore diameter of no more than 2.5 μm , and a strength of at least 100 MPa.

32. The ceramic of claim 31 wherein between about 60 wt% and 70 wt% of the silicon carbide comprises fine grains having a grain size of no more than 5 μm .

35 33. The ceramic of claim 28 wherein the wafer is unground.

34. The ceramic of claim 33 having a thickness of between 0.1 mm and 0.3 mm.
35. The ceramic of claim 28 wherein the wafer has a D_{50} pore diameter of at least about 1 μm .
- 5 36. The wafer of claim 28 wherein between 30 wt% and 50 wt% of the SiC comprises coarse grains having a grain size of at least 30 μm .
37. A process for making high strength, tape cast SiC wafers, comprising the steps of:
- 10 a) providing a formulation consisting essentially of silicon carbide powder and water, wherein:
- i) between 50 wt% and 70 wt% of the SiC powder has a particle size of no more than 5 μm , and
- ii) between 30 wt% and 50 wt% of the SiC powder has
- 15 a particle size of at least 20 μm ,
- b) tape casting the formulation to produce a tape cast body,
- c) drying the tape cast body to produce a green body having a thickness of between about 0.1 mm and 0.6
- 20 mm, and
- d) sintering the green body at a temperature of no more than 1850°C,
- to form a recrystallized silicon carbide wafer having a density of no more than 2.35 g/cc and a strength of
- 25 at least 80 MPa.
38. The process of claim 37, wherein the sintering temperature is no more than 1750°C, and the recrystallized silicon carbide wafer has a D_{95} pore diameter of no more than 2.5 μm and a strength of at least 90 MPa.
- 30 39. The process of claim 37 wherein the SiC powder comprises at least 60 wt% SiC particles having a size of no more than 5 μm , and the recrystallized silicon carbide wafer has a D_{95} pore diameter of no more than 3 μm .
- 35 40. The process of claim 37, wherein the sintering temperature is no more than 1750°C, the SiC powder comprises at least 60 wt% SiC particles having a size of no more than 5 μm ,

and the recrystallized silicon carbide wafer has D_{85} pore diameter of no more than 2.5 μm , a density of no more than 2.25 g/cc and a strength of at least 100 MPa.

41. The process of claim 37, wherein the sintering temperature
5 is no more than 1700°C, the SiC powder comprises at least 65 wt% SiC particles having a size of no more than 5 μm , and the recrystallized silicon carbide wafer has a strength of at least 103 MPa.

42. The process of claim 37 including the additional step of
10 compacting the tape cast body to reduce its thickness by at least 5% prior to the step of sintering.

43. A process for making high strength, tape cast ceramic wafers, comprising the steps of:

a) providing a formulation consisting essentially of a
15 ceramic powder, binder and water,

b) tape casting the formulation to produce a tape cast body,

c) drying the tape cast body to produce a green body having a thickness,

20 d) compacting the green body to reduce its thickness by at least 5%, and

e) sintering the green body.

INTERNATIONAL SEARCH REPORT

Internat Application No

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A. CLASSIFICATION OF SUBJECT MATTER
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According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C04B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X,P	WO 96 26910 A (SAINT-GOBAIN/NORTON INDUSTRIAL CERAMICS CORPORATION) 6 September 1996 see page 8, line 15 - line 24; claims 1-25,31-40	1-8, 20-36
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A	EP 0 240 160 A (IMPERIAL CHEMICAL INDUSTRIES PLC) 7 October 1987 see page 3, line 8 - line 11; claims 1-4,10	9,18,37, 43

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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INTERNATIONAL SEARCH REPORT

Information on patent family members

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